



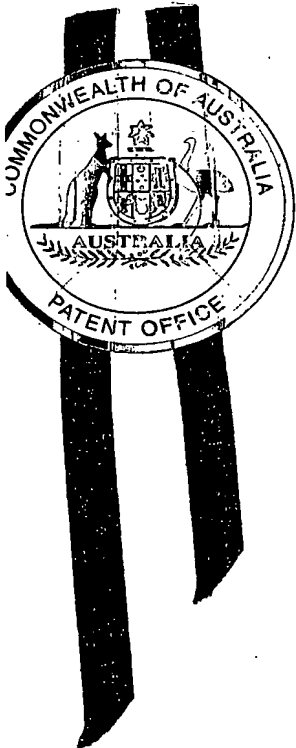
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I, JULIE BILLINGSLEY, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2002950815 for a patent by WMC RESOURCES LTD as filed on 15 August 2002.



WITNESS my hand this  
Twenty-second day of August 2003

*J. Billingsley*

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AUSTRALIA  
Patents Act 1990

PROVISIONAL SPECIFICATION

Applicant(s):

WMC RESOURCES LTD

Invention Title:

RECOVERY NICKEL

The invention is described in the following statement:

RECOVERING NICKEL

5           The present invention relates to a process for recovering valuable metals, such as nickel, from liquors obtained by processing laterite ores and concentrates that are contaminated with high levels of iron.

10           The term "processing laterite ores and concentrates" is understood herein to include processing by any one or more of heap leaching, pressure leaching, bacterial oxidation leaching, and atmospheric tank leaching.

15           The present invention relates particularly, although by no means exclusively, to a process for recovering nickel and cobalt from liquors obtained by acid leaching ores and concentrates that are contaminated with  
20 high levels of iron.

          In this context, the term "high levels of iron" is understood to mean levels of iron whereby the mole ratio of Fe:Ni is greater than 2:1.

25           It is known that nickel can be recovered from such liquors containing the nickel by contacting the liquors with  $H_2S$  to precipitate nickel sulphides (and mixed sulphides in situations where nickel and other valuable  
30 metals such as cobalt are in the liquors).

          However, it is also known that iron will co-precipitate as a sulphide under conventional  $H_2S$  precipitation conditions and that such co-precipitation is  
35 undesirable from the viewpoint of optimising recovery of nickel (and cobalt).

It is known that iron can be removed from liquors prior to nickel (and cobalt) precipitation by using (a) high temperatures (180-220°C) to selectively precipitate iron as hematite (b) low temperatures (90-120°C) to precipitate iron as goethite, and low temperatures (70-150°C) to precipitate iron as jarosite. However, high temperature precipitation is capital intensive, requiring autoclaves and flash vessels, and low temperature precipitation results in high nickel losses as a result of nickel adsorption onto iron species.

The applicant has developed a process that is capable of recovering very high levels (greater than 99%) of nickel from nickel liquors with very low levels of co-precipitation of iron.

According to the present invention there is provided a process for recovering valuable metals from laterite ores and concentrates that are contaminated with iron, which process includes the steps of:

- (a) reducing ferric ions to ferrous ions in a leach liquor containing a valuable metal and iron in solution using a suitable reductant ;
- (b) neutralising the liquor to reduce the free acid concentration in solution to levels suitable for nickel precipitation; and
- (c) precipitating the valuable metal using the reductant and seed particles under process conditions including one or more of seed particle size, seed composition, and temperature that are selected to maximise nickel precipitation and to minimise iron precipitation.

Preferably reduction step (a) includes reducing ferric ions to ferrous ions using the reductant in the

presence of 40-90 g/l free acid.

The reductant may be any suitable reductant.

5           One suitable reductant is NaHS.

Preferably the reductant is a gaseous reductant.

Preferably the gaseous reductant is  $H_2S$ .

10           Preferably neutralisation step (b) increases the  
pH of the solution to 2.

15           Preferably neutralisation step (b) maintains iron  
in the ferrous state.

Preferably the valuable metal is nickel.

20           More preferably the valuable metals are nickel  
and cobalt.

Preferably the laterite ores are ores that  
contain nickel in a chlorite mineral phase.

25           Preferably the process conditions for  
precipitation step (c) include operating at a partial  
pressure of the gaseous reductant of less than 60 psi.

30           More preferably the gas partial pressure is less  
than 40 psi.

More preferably the gas partial pressure is less  
than 30 psi.

35           It is preferred particularly that the gas partial  
pressure be less than 25 psi.

Preferably the process conditions for precipitation step (c) include operating at a liquor temperature of at least 50°C.

5           More preferably the liquor temperature is at least 60°C.

Preferably the seed particles for precipitation step (c) have a particle size of  $P_{50}$  less than 100 micron.

10

More preferably the particle size of the seed particles is  $P_{50}$  less than 80 micron.

15           It is preferred particularly that the particle size of the seed particles be  $P_{50}$  less than 60 micron.

Preferably the seed particle concentration for precipitation step (c) is greater than 30g/l.

20           More preferably the seed particle concentration is greater than 40g/l.

25           Preferably the ratio of iron and the valuable metal in the leach liquor supplied to step (a) is greater than 2:1.

More preferably the ratio is greater than 3:1.

More preferably the ratio is greater than 5:1.

30

The present invention is based on extensive experimental work carried out by the applicant to recover nickel and cobalt from laterite ores. The experimental work included the following work.

35

#### EXPERIMENTAL

Initial sulphide precipitation testwork using a gaseous reductant in the form of  $H_2S$  gas in the presence of seed particles was carried out on synthetic liquor generated by dissolving technical grade sulphate salts in tap water.

The initial testwork included testwork for optimal precipitation conditions using the synthetic liquor.

The initial testwork investigated the following precipitation parameters to determine their effect on nickel and cobalt recovery and nickel/iron separation:

- Temperature
- $H_2S$  pressure
- Seed particle size
- Seed concentration,

The initial testwork for optimal precipitation conditions was conducted under the following conditions:

- Agitation = 750 rpm with twin axial turbines
- Sampling time = 15, 30 and 60 minutes (in some instances a 120 minute sample was also taken).
- Seed chemistry = synthetic  $NiS$ ,  $Ni:S = 0.95$

Once the optimal conditions for sulphide precipitation were determined, further tests were conducted using liquors generated from heap leaching of laterite ores from a range of different sources. The different sources are referred to as GA-Ev, GA-Tr, GL-Ka, and GL-Ak in the following description.

The heap leach liquors were spiked with nickel and cobalt sulphate salts to increase the nickel and cobalt concentration in solution to ~4 and 0.2 g/l respectively to simulate recycling of liquor to the heap.

Analysis of the liquors used in the testwork is given in Table 1.

5                      Table 1 - Liquor Compositions

| Liq. Source | <sup>D</sup> Ni | Co  | <sup>D</sup> Fe | <sup>D</sup> Mg | Cr  | <sup>D</sup> Al | Mn  | Cu | Zn | <sup>D</sup> FA |
|-------------|-----------------|-----|-----------------|-----------------|-----|-----------------|-----|----|----|-----------------|
| Synthetic   | 4.04            | 198 | 24.9            | 5.94            | 526 | 8.00            | 706 | 1  | 5  | 1.0             |
| GA-Ev       | 4.16            | 204 | 26.0            | 4.58            | 593 | 4.46            | 470 | 5  | 5  | 80              |
| GA-Tr       | 4.02            | 196 | 22.6            | 3.62            | 749 | 3.91            | 511 | 4  | 3  | 86              |
| GA-Ka       | 4.20            | 200 | 16.4            | 13.4            | 108 | 0.272           | 313 | 3  | 73 | 45              |
| GA-Ak       | 4.25            | 210 | 27.2            | 4.82            | 518 | 4.88            | 455 | 3  | 3  | 73              |

Assays are in mg/L  
D denotes assays in g/L

- 10                      Sulphide product assays were all back calculated to discount the seed component.

### RESULTS

15      Effect of Temperature on Mixed Sulphide Precipitation

Temperature influences the precipitation process through its influence on H<sub>2</sub>S solubility and the dissociation equilibrium constant for H<sub>2</sub>S.

- 20                      The kinetics of precipitation are also influenced by temperature.

- 25                      The effect of temperature on metal precipitation was determined under the following conditions:

- |                           |   |             |
|---------------------------|---|-------------|
| Seed P <sub>50</sub>      | - | 109 microns |
| H <sub>2</sub> S pressure | - | 30 psi      |
| Seed concentration        | - | 30 g/L      |
| 30      Residence Time    | - | 60 minutes  |

The detailed results for Tests 1 to 4 from these tests are summarised in Figure 1.



The results show that increasing temperature increased metal precipitation. Metal precipitation increased dramatically between 40°C and 60°C while between 80°C and 95°C only a marginal increase in metal precipitation was observed after 60 minutes. The Ni:Fe mole ratio ranged from 7 to 10 between 60 and 95°C and appears to reach a maximum at 80°C and then decrease again at 95°C.

Precipitation of aluminium, magnesium, manganese and chromium were low (<1%) under the conditions investigated.

#### 15 Effect of Seed Charge

In commercial operations seed particles are added to facilitate precipitation. It is expected that as the seed surface area increases the kinetics of precipitation increases, thereby resulting in a lower  $S^{2-}$  concentration in solution, and enabling better selectivity for nickel and cobalt over iron.

The surface area of the seed can be increased by increasing the mass of seed in the reactor or by seeding with solids with a smaller average particle size. The influence of both factors was investigated. Detailed results are summarised in Figures 2 and 3.

The results show that nickel and cobalt recovery increased with the mass of seed added and with a decreasing average seed particle size. Both these observations are consistent with nickel and cobalt recovery increasing with increasing seed surface area.

When the seed particle size was reduced from  $P_{50} = 109$  microns to  $P_{50} = 45$  microns nickel recovery increased

from 98.2 to 99.5% while cobalt recovery increased from 99.0 to 99.7%.

Using the finer seed had little effect on the Ni:Fe mole ratio in the product which appeared to decrease slightly from 7.0 to 6.7. This change in the Ni:Fe ratio is probably not significant within the errors of the experiment.

When the seed concentration was increased from 30 to 50g/l a similar increase in nickel and cobalt recoveries was observed. The Ni:Fe mole ratio in the product however decreased from 7.0 to 4.7 due to iron precipitation increasing from 2.2 to 3.2%. These results are inconsistent with those observed with the finer seed. A possible explanation is that the test using the higher seed concentration (Test 10) was conducted significantly later than all the other tests and oxidation of the ferrous in the feed liquor appears to have resulted in a higher ferric concentration in this solution relative to the previous tests. This is supported by the higher free acid concentration in solution at the end of Test 10 (~17g/l as opposed to ~8g/l previously) and the higher sulphur concentration (62% S as opposed to 48% S) in the sulphide product. Both these observations are consistent with  $H_2S$  reacting with  $Fe(III)$  in solution according to the following reaction.



The above reaction produces free acid and elemental sulphur as observed in test 10. The presence of  $Fe(III)$  in solution would also be expected to have resulted in higher iron precipitation, reducing the Ni:Fe in the sulphide product.

### Effect of Hydrogen Sulphide Pressure

Previous testwork indicated that H<sub>2</sub>S pressure between 30 and 60 psi had a strong influence on metal precipitation. In the current testwork the effect of H<sub>2</sub>S pressure was investigated further.

Precipitation tests were conducted at 80°C, 30 g/l seed, with a P<sub>50</sub> of 109 microns. The results are presented in Figure 4.

The results show nickel recovery increased from 97.1 to 99.8% while cobalt recovery increased from 96.8 to 99.7% as H<sub>2</sub>S pressure increased from 20 to 40 psi while the Ni:Fe mole ratio of the product decreased from 11.3 to 6.6. This corresponds to iron precipitation increasing from 1.3 to 2.3%.

These results are all consistent with S<sup>2-</sup> concentration in solution increasing with H<sub>2</sub>S partial pressure resulting in greater metal recovery and lower selectivity.

### Column Heap Leach Liquors

From the above series of tests the following optimal conditions required for precipitating nickel and cobalt from heap leach liquors were determined:

- |                             |             |
|-----------------------------|-------------|
| • H <sub>2</sub> S Pressure | 20 psi      |
| • Seed charge               | 30 g/l      |
| • Temperature               | 80°C        |
| • Seed P <sub>50</sub>      | 45 microns. |

Initially heap leach liquors were partially neutralised to remove excess free acid and then treated with H<sub>2</sub>S to precipitate nickel and cobalt without a pre-

reduction stage.

This resulted in low nickel and cobalt precipitation and the product was contaminated with iron and elemental sulphur. In addition, the liquor free acid also increased sharply, according to reaction 1.

A second batch of liquors was generated and treated in the following sequence of process stages, as discussed below:

- Pre-reduction
- Neutralisation
- Sulphide Precipitation

#### Pre-Reduction Stage

The following pre-reduction stage was selected with the objective of reducing all Fe(III) to Fe(II):

Temperature                      80°C  
H<sub>2</sub>S pressure                      20 psi  
Reaction time                      90 minutes  
Initial free acid                  40-90 g/l H<sub>2</sub>SO<sub>4</sub>

The results are summarised in Table 2.

Table 2 - Results of Pre-Reduction with H<sub>2</sub>S

| Sample | % Precipitation |       |    |      |       | Final Aq. Assays (g/L) |      |      |
|--------|-----------------|-------|----|------|-------|------------------------|------|------|
|        | Ni              | Co    | Cu | Zn   | Cr    | Fe(II)                 | Fe   | F.A  |
| GL-Ev  | 0.25            | 0.01  | 94 | 0.24 | 0.02  | 26.7                   | 26.7 | 101  |
| GL-Tr  | 0.67            | 0.01  | 87 | 0.46 | 0.02  | 22.2                   | 22.1 | 104  |
| GL-Kaa | 0.08            | 0.01  | 81 | 0.01 | 0.003 | 16.7                   | 17.3 | 58.2 |
| GL-Ak  | 0.13            | 0.005 | 76 | 0.30 | 0.12  | 26.4                   | 26.6 | 95   |

The Fe(III) was effectively reduced to Fe(II)

under the conditions employed.

Nickel precipitation ranged from 0.083 to 0.67 while cobalt losses were less than 0.01%.

5

Copper was the only element which showed significant precipitation with copper concentrations in solution reduced to less than 1 mg/l.

10

The concentrations of the other elements assayed (Fe, Zn, Cr, Mg, Mn and Al) were not significantly altered by the pre-reduction stage with precipitation consistently less than 0.5% and typically less than 0.1%.

15

As a result of the Fe(III) reduction the free acid concentration increased and elemental sulphur was generated.

20

The observed increase in free acid concentration in solution was in good agreement with the calculated increase in free acid concentration based on reaction 1 stoichiometry.

25

Residue assays showed sulphur as the main constituent (>80%).

30

The results show that the Fe(III) could be effectively reduced to Fe(II) using  $H_2S$  in the presence of relatively high free acid (40-90 g/l) with minimal nickel and cobalt losses.

#### Neutralisation Stage

35

The liquors generated from the pre-reduction stage were treated with limestone to increase the solution pH to -2 in the neutralisation stage.

The feed liquor compositions supplied to the neutralisation stage are given in Table 3.

Table 3 - Liquor Compositions to Neutralisation Stage

5

| Liq. Source | <sup>D</sup> Ni | Co  | <sup>D</sup> Fe | <sup>D</sup> Mg | Cr  | <sup>D</sup> Al | Mn  | Cu  | Zn | <sup>D</sup> FA |
|-------------|-----------------|-----|-----------------|-----------------|-----|-----------------|-----|-----|----|-----------------|
| GL-Ev       | 4.27            | 210 | 26.7            | 4.80            | 622 | 5.12            | 497 | 0.4 | 5  | 101             |
| GL-Tr       | 4.07            | 197 | 22.1            | 3.83            | 758 | 4.32            | 519 | 0.5 | 3  | 104             |
| GL-Ka       | 4.43            | 197 | 17.3            | 14.2            | 119 | 0.347           | 330 | 0.6 | 77 | 58              |
| GL-Ak       | 4.14            | 196 | 26.6            | 4.94            | 565 | 5.45            | 470 | 0.7 | 3  | 95              |

Assays are in mg/L  
D denotes assays in g/L

10 The liquors were neutralised by being treated with limestone ( $P_{80}=250$  microns) at ambient temperature. The results of the neutralisation tests are summarised in Table 4.

Table 4 - Results Of Liquor Neutralisation

15

| Sample | % Precipitation |      |      |     |     | Final Aq. Assays (g/L) |      |    |
|--------|-----------------|------|------|-----|-----|------------------------|------|----|
|        | Ni              | Co   | Fe   | Zn  | Cr  | Fe(II)                 | Fe   | pH |
| GL-Ev  | 0.37            | 0.07 | 0.11 | 3   | 0.1 | 26.8                   | 27.9 | 2  |
| GL-Tr  | 0.40            | 0.16 | 0.01 | 14  | 1   | 22.0                   | 22.5 | 2  |
| GL-Ka  | 0.26            | 0.05 | 0.17 | 0.2 | 4   | 15.6                   | 17.3 | 2  |
| GL-Ak  | 0.33            | 0.15 | 0.10 | 9   | 2   | 26.1                   | 28.3 | 2  |

20 Nickel and cobalt losses by precipitation were low - ranging from 0.2 to 0.4% and 0.05% to 0.16%, respectively.

The copper concentration in solution was reduced to below the copper detection limit of 0.1 mg/l.

25 Precipitation of the remaining impurities (Al, Mn, Cr, Fe and Zn) was low, generally less than 1%.

30 The iron essentially remained in the Fe(II) state enabling sulphide precipitation on the liquor to be undertaken.

The residues were mainly composed of gypsum (indicated by high calcium and sulphur assays).

5 Limestone consumption was very high, ranging from 13 to 24 kg/kg (Ni+Co) - due to the high free acid concentration in the solution.

10 Overall nickel and cobalt losses across pre-reduction and neutralisation stages ranged from 0.3 to 1.1% and 0.06 to 0.17%, respectively.

#### Sulphide Precipitation Stage

15 Following the pre-reduction and neutralisation stages the clarified liquors were treated with H<sub>2</sub>S to precipitate nickel and cobalt under the following conditions.

- 20
- H<sub>2</sub>S Pressure 20 psi
  - Seed charge 30 g/l
  - Temperature 80°C
  - Seed P<sub>50</sub> 45 microns.

25 The composition of the liquors used in the sulphide precipitation stage are summarised in Table 5.

Table 5 - Liquor Compositions to  
Sulphide Precipitation Stage

30

| Sample | Liquor Assay (mg/L) |     |       |     |     |      |     |    |       |
|--------|---------------------|-----|-------|-----|-----|------|-----|----|-------|
|        | Ni                  | Co  | Fe    | Cu  | Cr  | Al   | Mn  | Zn | Mg    |
| GL-Ev  | 4250                | 210 | 27940 | 0.2 | 621 | 5080 | 497 | 5  | 4780  |
| GL-Tr  | 4050                | 197 | 22510 | 0.2 | 750 | 4260 | 519 | 3  | 3810  |
| GL-Ka  | 4420                | 197 | 17280 | 0.2 | 114 | 347  | 330 | 77 | 14220 |
| GL-Ak  | 4130                | 196 | 28340 | 0.4 | 554 | 5410 | 470 | 3  | 4930  |

The results of sulphide precipitation are

summarised in Table 6.

Table 6 - Results Of Sulphide Precipitation

| Liquor | % Precipitation |      |     | Solids Assays (%) |     |     |      |      |       |      | Solids<br>#Ni:Fe |
|--------|-----------------|------|-----|-------------------|-----|-----|------|------|-------|------|------------------|
|        | Ni              | Co   | Fe  | Ni                | Co  | Fe  | Cr   | Al   | Zn    | S    |                  |
| GL-Ev  | 99.2            | 99.0 | 1.7 | 47.6              | 2.3 | 5.0 | 0.13 | 0.05 | 0.03  | 30.8 | 9.0              |
| GL-Tr  | 99.3            | 99.8 | 1.9 | 47.5              | 2.5 | 4.3 | 0.08 | 0.03 | 0.01  | 29.7 | 10.6             |
| GL-Ka  | 99.3            | 99.6 | 2.7 | 48.7              | 2.3 | 4.6 | 0.02 | 0.01 | 0.73  | 31.3 | 10.1             |
| GL-Ak  | 99.4            | 99.8 | 1.3 | 47.0              | 2.4 | 4.0 | 0.10 | 0.04 | <0.01 | 32.1 | 11.2             |

5 # Mole Ratio

Despite the relatively low H<sub>2</sub>S partial pressure, nickel and cobalt recoveries were high (>99%).

10 In the optimisation work the same H<sub>2</sub>S pressure and temperature yielded recoveries of ~97% for nickel and cobalt.

The higher recoveries observed with the heap  
15 leach solutions is attributed to using a finer seed P<sub>50</sub>=45 microns compared to P<sub>50</sub>=109 microns for the synthetic solutions. The sulphate levels were similar for synthetic and real liquors (~100 g/l SO<sub>4</sub><sup>2-</sup>) and were not expected to have resulted in significant differences in nickel and  
20 cobalt precipitation.

The solids contained between 4.0 and 5.0% Fe with the Ni:Fe mole ratio ranging between 9.0 and 11.2. These results are in good agreement with the results  
25 obtained under similar conditions in the optimisation testwork. The results showed that the Ni:Fe mole ratio could be upgraded from a range of 0.14-0.24 in solution to 9.0-11.2 in the sulphide product representing approximately a 50 fold upgrade of the nickel values with  
30 respect to iron.

Aluminium and chromium showed increased



precipitation as the concentration of these metals in solution increased. However the concentrations of aluminium and chromium in the product remained low. These results are summarised in Figure 5.

5

The aluminium concentration in the product generally increased with aluminium concentration in solution. Aluminium does not form stable sulphides in solution and the increase in aluminium concentration in the product appears to be through adsorption/entrainment with the sulphide.

10

The chromium concentration in the product also increased with chromium concentration in solution. This effect is much stronger with the increase in chromium in the product increasing more steeply with chromium in the PLS compared to aluminium and could indicate that chromium precipitated as opposed to being adsorbed/entrained.

15

Zinc concentration in solution was reduced to between 2 and 3 mg/l. The sulphide products were generally low in zinc (less than 0.03%) due to the relatively low zinc concentration in solution (3-5 mg/l). Only the sulphide generated from the GL-Ka ore contained significant zinc at 0.73% due to the feed solution having a higher zinc tenor (77 mg/l).

20

25

Copper concentration in the products was low due to copper removal in previous unit operations with copper in the product ranging between 0.01 and 0.03%.

30

Manganese and magnesium concentrations in the products were consistently low at less than 50 ppm and less than 500 ppm, respectively, reflecting the high selectivity of sulphide precipitation against these elements.

35

### CONCLUSIONS

From the optimisation testwork the following conclusions can be drawn.

5

1. Nickel and cobalt precipitation increased as liquor temperature increased from 40°C to 95°C. The Ni/Fe separation appeared to reach a maximum at 80°C. Aluminium, chromium, manganese and magnesium precipitation were not affected over the temperature range investigated.

10

2. Increased seed surface area increased nickel and cobalt recovery.

15

3. Increasing the partial pressure of H<sub>2</sub>S from 20 to 40 psi increased nickel recovery from 97.1 to 99.8% and the cobalt recovery from 96.8 to 99.7%. The Ni:Fe mole ratio in the product decreased from 11 to 6.7.

20

From the testwork conducted on the heap leach liquors the following conclusions can be drawn.

1. Liquors can be treated with H<sub>2</sub>S gas in a pre-reduction stage to reduce Fe(III) to Fe(II) with low nickel and cobalt losses (ranging from 0.08% - 0.67% and <0.1%, respectively). Copper was the only impurity significantly removed during pre-reduction with copper concentration in solution being reduced below 1 mg/l.

25

30

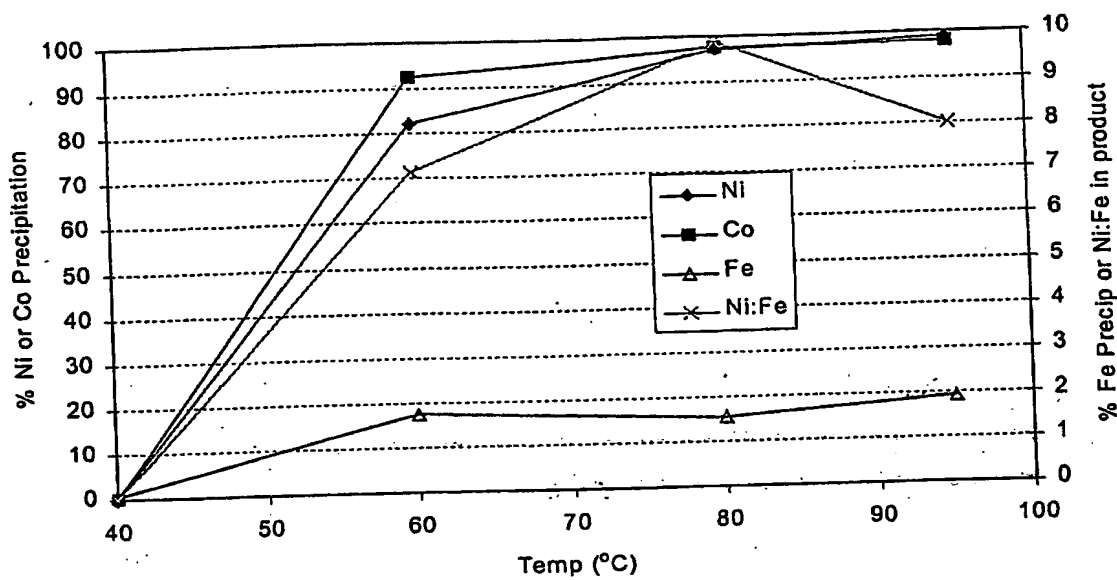
2. The neutralisation stage effectively increased the pH of the liquor from the pre-reduction stage to ~2 while maintaining iron in the Fe(II) state. Nickel and cobalt losses during the neutralisation stage were 0.2%-0.4% and 0.05%-0.16%, respectively. The

35

residues were mainly gypsum. Impurity removal during neutralisation was generally less than 1%.

5        3. Sulphide precipitation was successful in precipitating greater than 99% of the nickel and cobalt, producing a precipitate with a Ni:Fe mole ratio between 9 and 11.2, from solutions containing ~ 4g/L nickel and 17-27 g/l iron. Chromium and to a lesser extent aluminium were present in the sulphide product in minor quantities. Zinc concentration in 10        solution was reduced to ~3 mg/l.

15        Many modifications may be made to the present invention described above without departing from the spirit and scope of the invention.



5

Figure 1: Ni, Co and Fe Precipitation After 1 hour verses Temperature

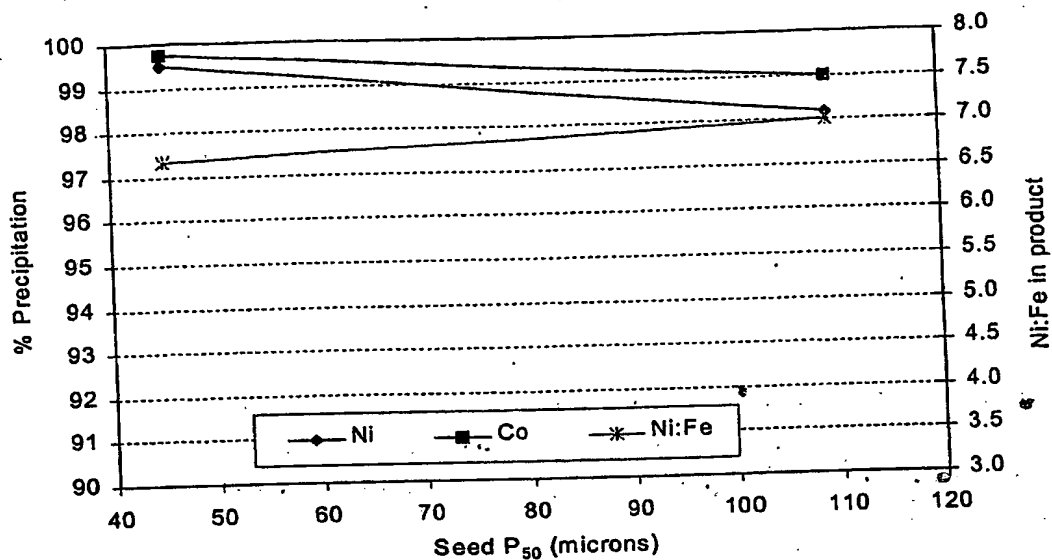


Figure 2: Effect of Seed P<sub>50</sub> size on Ni and Co Precipitation and Ni:Fe Ratio in the Sulphide Product.

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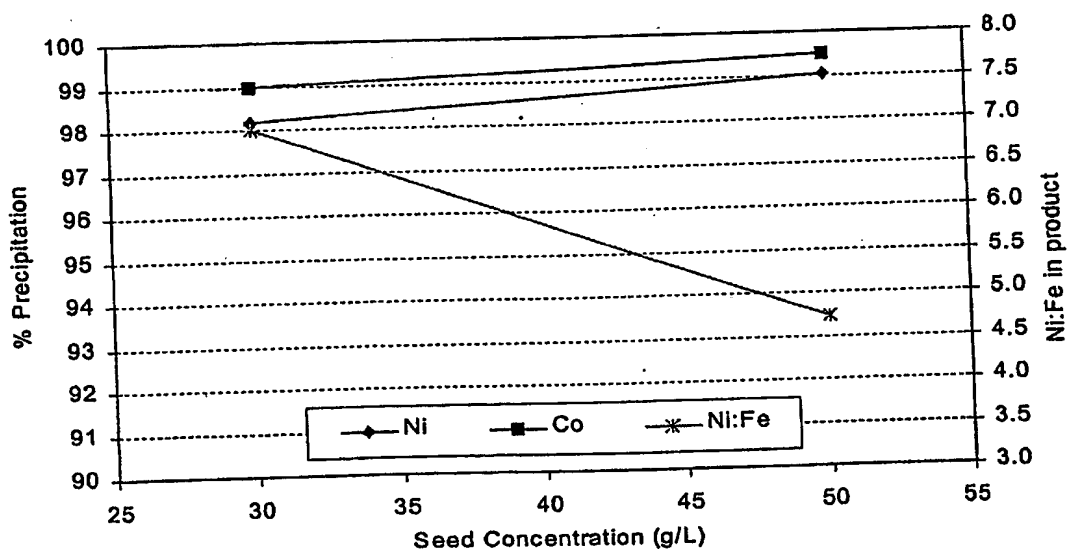


Figure 3 - Effect of Seed Concentration on Ni and Co Precipitation and Ni:Fe Ratio in the Sulphide Product

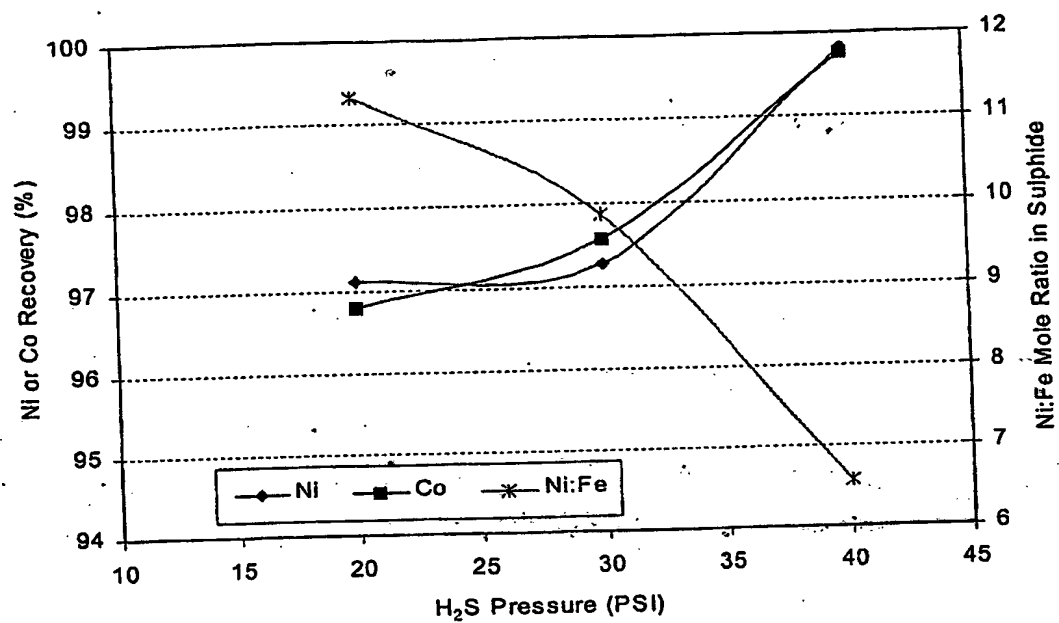
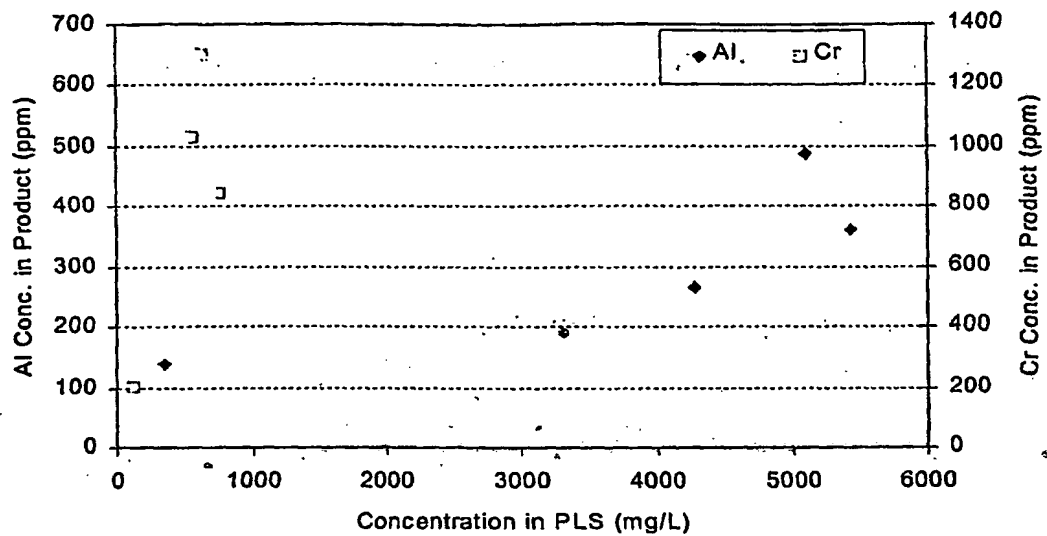


Figure 4 – Effect of H<sub>2</sub>S Partial Pressure on Ni, Co Recovery and Ni:Fe Mole Ratio in the Product

5



5

**Figure 5 – Affect of Al and Cr Concentration in Solution on Al and Cr Concentration in the Sulphide**